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Chapter 2

LEAD IN SOIL BY FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY— AN EXAMINATION OF PAIRED IN-SITU AND LABORATORY ICP-AES RESULTS

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ABSTRACT

A major aspect of lead hazard control is the evaluation of soil lead hazards around housing coated with lead-based paint. The use of field-portable X-ray fluorescence (FPXRF) to do detailed surveying, with limited laboratory confirmation, can provide lead measurements in soil (especially for planning abatement activities) in a far more cost-efficient and timely manner than laboratory analysis. To date, one obstacle to the acceptance of FPXRF as an approved method of measuring lead in soil has been a lack of correspondence between field and laboratory results. In order to minimize the differences between field and laboratory results, RTI International (RTI) has developed a new protocol for field drying and sieving of collected samples for field measurement by FPXRF. To evaluate this new protocol, composite samples were collected in the field following both HUD Guidelines and American Society for Testing and Materials (ASTM) protocols, measured after drying by FPXRF, and returned to the laboratory for confirmatory inductively coupled plasma atomic emission spectroscopy (ICP-AES) analysis. Evaluation of study data from several diverse sites revealed no statistical difference between paired FPXRF and ICP-AES measurements using the new method.

Keywords: lead, soil, XRF, ICP-AES, HUD, field

1. INTRODUCTION

Two major aspects of lead hazard control are the evaluation and mitigation of soil lead hazards around housing that is coated in part with lead-based paint or that exhibits lead contamination from other sources. Major sources of lead in soil include lead-based paint on

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exterior surfaces that have deteriorated, allowing the lead from the paint to leach into the drip line soil, or organo-lead from automobile exhaust that has been deposited as ultra-fine metal halide aerosols directly onto the soil or onto other surfaces and then leached into the soil. Lead-containing soil may be ingested by children when they play outdoors. It may also be tracked into the house and collect as dust on floors, window sills, toys, utensils, etc., and be ingested through hand-to-mouth activities or inhaled as dust (Mielke and Reagan, 1998). Lead, even at low levels, can cause central nervous system impairment, mental retardation, and behavioral disorders (Needleman et al., 1990).

Recent advances in analytical measurement and sampling design for lead in soil offer significant opportunities for improving soil testing methodology. Using FPXRF to do detailed surveying, with limited laboratory confirmation, can provide more cost-efficient and timely lead measurements in soil (especially for planning abatement activities) than laboratory analysis can. U.S. Environmental Protection Agency (USEPA) Method 6200 provides an approved FPXRF screening method for 26 analytes (including lead) in soil and sediment (USEPA, 1998).

Several groups have reported on the successful use of FPXRF for soil-lead measurement. A pilot study of sources of lead exposure in residential settings was conducted in a mining and smelting district in northern Armenia. A multi-element XRF analyzer was used to test for lead in soil. The highest lead levels were found in loose exterior dust samples, and lead concentrations in yard soil were higher than those in garden soil (Petrosyan et al., 2004). In another study, lead in soil adjacent to an urban highway was measured using FPXRF. Lead content in soil samples collected 15 feet from the highway was determined to be greater than 2,000 ppm. Soil lead decreased as the perpendicular distance from the highway increased (Bachofer, 2004). Lead in soil was tested at 11 San Francisco area houses. FPXRF readings were significantly correlated ($p < 0.0005$) with laboratory results and met the study criteria for an acceptable screening method (Reames and Lance, 2002). Although this study and others have shown a correlation between field and laboratory results, the lack of a 1:1 correspondence has essentially hindered the practical application of field XRF measurements.

Several previous studies have indicated that if you provide a field sample similar in particle size and dryness to the prepared laboratory confirmatory sample, a near 1:1 correspondence can be obtained when comparing FPXRF to ICP-AES (Maxfield, 2000). In a study comparing field FPXRF values measured in situ on soils in Poland, geometric mean soil lead concentrations were 200 ppm with the portable XRF and 190 ppm using atomic absorption, with excellent correlation for samples sieved to less than 250μ ($p = 0.0001$) (Clark et al., 1999). Another study concluded that "the best results were achieved when the soil samples were prepared prior to their FPXRF analysis" (Boyle and Fitzgerald, 2004). Previous work conducted by RTI has shown a near 1:1 correlation between prepared (dried, ground, sieved) samples measured by both FPXRF and ICP-AES (Binstock and Gutknecht, 2002). Ideally, each field sample would be dried and sieved for FPXRF measurement. The question is how to accomplish this in the field in a cost-efficient manner.

RTI has developed a new protocol for field drying and sieving of collected samples for measurement by FPXRF. This protocol follows traditional HUD and ASTM sampling protocols for housing, with field measurement by FPXRF of the collected samples. In order to evaluate this protocol, composite samples were collected following both HUD Guidelines and ASTM

protocols, measured after drying and sieving by FPXRF, and returned to the laboratory for confirmatory ICP-AES analysis.

2. MATERIAL AND METHODS

Soil sampling and measurement of soil lead in residential yards was performed in six cities across the United States. Site locations ranged from Charlotte, NC, to Minneapolis, MN (Table 1). At several potential sites in each city, FPXRF screening analysis was performed in situ, and the two most suitable sites (based on lead levels, accessibility, and size of drip line) were chosen for the study. Screening analysis involved taking several 30-second surface XRF readings at locations along the drip line and from any bare play areas around the dwelling to gain an estimate of soil lead levels. The instrument used was a Niton XL-309 equipped with a 10mCi Cd-109 source.

Table 1. Site Locations for FPXRF Sampling

Site Location	ID	Year Built
Greensboro, NC	NCG-1	1931
	NCG-2	1922
Rochester, NY	NY-1	1925
	NY-2	1920
Knoxville, TN	TN-1	1945
	TN-2	1947
Charlotte, NC	NCC-1	1922
	NCC-2	1929
Petersburg, VA	VA-1	1940
	VA-2	1917
Minneapolis, MN	MN-1	1900
	MN-2	1900

Following HUD Guidelines protocol for soil sampling, at least two composite samples were collected from each drip line area (HUD, 1995). Each composite was composed of five individual 0.5-inch cores; each core was taken from an area at least 2 feet from another core and 2 feet from the dwelling foundation. All cores were collected using a 10-gram Terra Core® single-use device sampler (En Novative Technologies, Inc., Green Bay, WI). In addition, at least two composites were collected from the same area following the ASTM standard practice for field collection of soil samples for lead determination (ASTM, 2000). Each composite was composed of three individual 0.5-inch deep cores collected from an area 2 feet from the dwelling foundation; one of the cores was collected at the center of this area, and two more cores from within a 1-foot diameter circle around this initial core.

All composite soil samples were placed in a 5-inch hexagonal weigh boat (VWR 25433-104), lightly pulverized with a glass rod to facilitate mixing, and dried. Average sample size was

approximately 75 grams. Depending on their moisture condition, samples were dried either by air (if slightly wet) or by a 700-watt microwave oven (GE JES738WJ) connected to a car battery using an inverter (Xantrex 1200 plus). If residential power was available, the microwave oven was connected directly to the house current. Drying typically required one 3-minute cycle at full microwave power. After being dried, the entire sample was placed on a 3-inch diameter stacked sieve composed of a 2mm screen atop a 250 μ screen, and shaken vigorously for 2 minutes. A stiff nylon bristle brush was used to clean the screens between samples (VWR 17210-008). Screened material of less than 250 μ was put into an XRF sample cup (Chemplex Industries No. 1330, Palm City, FL) and analyzed in duplicate by FPXRF using a 30-second exposure time (Niton XL-309 instrument).

Dried and sieved samples were shipped to the laboratory for acid digestion and ICP-AES analysis (Binstock et al., 1997). In the laboratory, a 0.2g portion of the sieved sample was removed from each XRF cup and placed into a 50mL centrifuge tube (BD Falcon 352098). Five mL of 25% HNO₃ was added and the centrifuge tubes immersed in an ultrasonic bath (Branson, model 5510) for 30 minutes. Upon removal of the tubes from the bath, deionized water was added to the 50mL mark. The samples were then shaken for 30 seconds and centrifuged for 20 minutes at 2,000 rpm. ICP-AES analysis was done using a Leeman Labs Prodigy high-dispersion ICP.

3. RESULTS

Table 2 presents mean FPXRF and mean ICP-AES results for all 12 sites. Statistical tests (sign test, Wilcoxon signed rank test, and paired t-test) on 11 of the 12 sites show no statistical difference between mean FPXRF and ICP-AES soil lead using HUD and ASTM sampling methods. The VA-1 site did not pass equivalency for the signed rank test.

Table 2. Results of FPXRF and ICP-AES Measurement of Soil Lead by Site

Site	Number of Composite Samples*	Mean XRF (mg Pb/Kg)	Mean ICP-AES (mg Pb/Kg)	R ²
NCG-1	4	2,660	2,800	0.92
NCG-2	8	1,620	1,690	0.99
NY-1	4	743	778	0.99
NY-2	4	960	974	1.00
TN-1	5	162	152	0.99
TN-2	6	291	298	0.98
NCC-1	6	3,180	3,070	0.98
NCC-2	6	399	337	1.00
VA-1	8	1,380	1,410	1.00
VA-2	8	828	845	1.00
MN-1	6	1,140	1,130	0.99
MN-2	7	3,490	3,590	0.99

*Collected using HUD and ASTM protocols

Regression analysis by sampling method over all sites similarly shows a near 1:1 correspondence between mean FPXRF and ICP-AES soil lead measurements with a slope of 1.02 for the HUD sampling method and a slope of 1.06 for the ASTM sampling method (Figures 1 and 2). Additionally, statistical tests (sign test, Wilcoxon signed rank test, and paired t-test) by sampling method over all sites shows no statistical difference between mean FPXRF and ICP-AES soil lead measurements.

Figure 1. FPXRF Pb of Dried, Sieved Soil vs. ICP-AES Pb of Acid-Digested Soil, HUD Sampling Method (N=35)

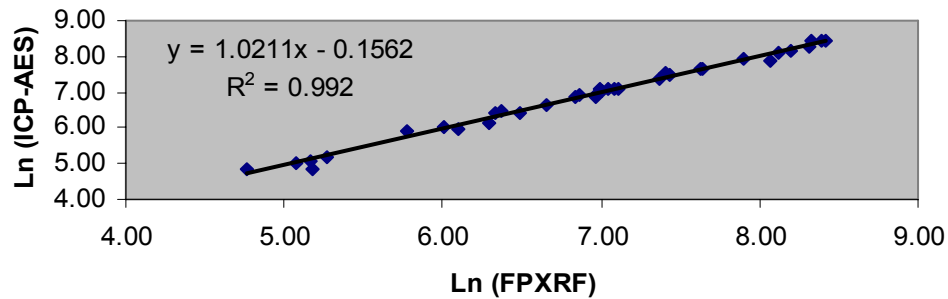
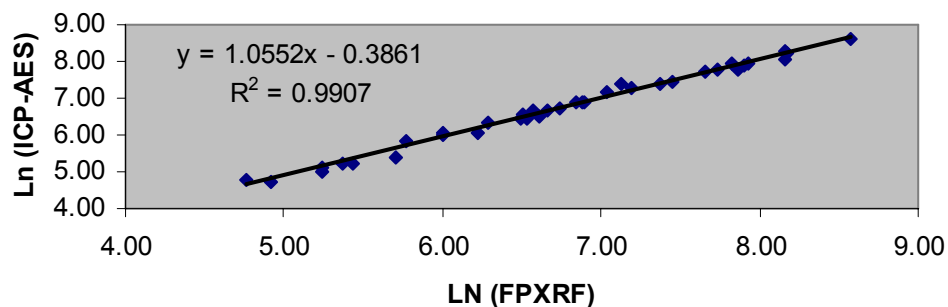


Figure 2. FPXRF Pb of Dried, Sieved Soil vs. ICP-AES Pb of Acid-Digested Soil, ASTM Sampling Method (N=37)



4. DISCUSSION/CONCLUSIONS

Examination of the data clearly shows a statistical equivalence for mean soil lead values of paired samples collected following HUD Guidelines and ASTM protocols and measured by FPXRF and laboratory ICP-AES values. The data is fairly extensive, comprising a total of 72 paired samples collected from 12 residential sites in 6 U.S. cities. A variety of soil types ranging from dry loam to sandy is represented. Each site presents a similar statistical equivalence between in situ FPXRF soil lead values and laboratory ICP-AES soil lead values.

The current approved methods of collecting residential soil samples used by lead inspectors and risk assessors—either HUD Guidelines, ASTM E1727-99 sampling protocol, or similar methods based on these, followed by shipment of the samples to an accredited laboratory for analysis—are fairly time consuming. In some cases, turnaround time for lab results can be as much as 2 weeks. In contrast, collecting, drying, sieving, and measuring composite samples in the field following current sampling protocol with FPXRF measurement can be done in less than 2 hours.

This research presents a strong case for the use of FPXRF methodology as a significant improvement over current protocols for sampling and analyzing lead in residential soils. With proper sample preparation, one can obtain results in the field that are not only statistically equivalent to those obtained in the laboratory, but also more timely and cost-efficient.

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